

FEU 05 – Distance Determination and Gunshot Residue Testing

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1. Scope

- 1.1. This standard operating procedure is designed to provide specific techniques for detecting and preserving patterns of vaporous and particulate lead and nitrite residues around a defect or impact as a basis for estimating muzzle-to-target distances.
- 1.2. The testing is limited and provides a likely range based on the comparison of the known test fired patterns and the patterns found on the questioned evidence.

2. Background

- 2.1. To establish the practices for documenting the examination of firearm evidence to conform to the requirements of the Department of Forensic Sciences (DFS) Forensic Science Laboratory (FSL) *Quality Assurance Manual*.

3. Safety

- 3.1. All Firearms Safety protocols in Appendix A will be adhered to when handling firearms.
- 3.2. Staff members should use universal precautions with evidentiary materials. Nitrile gloves will be utilized when performing chemical testing, including sodium rhodizonate and Griess tests.
- 3.3. All chemical testing, to include mixing of chemicals, requires the use of a disposable lab coat and eye protection.

- 3.4. Mixing of chemicals requires the use of a disposable lab coat, worn so that no skin is exposed between the coat sleeves and gloves, and safety goggles; additionally the use of an N95 respirator mask is recommended.
- 3.5. Ensure the proper ventilation is provided during both the preparation of the reagents and the procedure itself.

4. Materials Required

- 4.1. Scissors, laboratory glassware, cheesecloth, tweezers, processing tray, flat iron, spatula, appropriate PPE, brown wrapping paper, photographic paper, exhaust hood, wire brush, filter paper, reagents: sulfanilic acid, sodium nitrate, glacial acetic acid, methanol, alpha-naphthol, concentrated hydrochloric acid, tartaric acid, sodium rhodizonate; distilled water, cotton swabs, re-sealable bags, balance, weigh boats, glass stir rod, magnetic stirrer, steel tape measure, stereo microscope, cotton twill cloth, felt marker, camera, lead and copper bullets, inertia bullet puller, tape and firing range.
- 4.2. On a muzzle to garment distance examination one must have the firearm in question and like ammunition.

5. Standards and Controls

- 5.1. Positive and negative controls are prepared for the Modified Griess test to detect nitrites. Nitrite cotton swabs are used for the positive control for Modified Griess Test.
- 5.2. A lead bullet is used as a positive control for the Sodium Rhodizonate Test (SoRo).

6. Calibration

- 6.1. Not applicable

7. Procedures

- 7.1. Visual and Macroscopic Examinations
 - 7.1.1. It will be noted that the initial examination is in regard to the macroscopically observable physical evidence characteristics and residues which may be present.
 - 7.1.2. Initially, a visual examination will be performed to determine the presence of bullet/pellet holes and gunshot residues. A sketch(s) or photograph(s) will be taken to document the item being examined and the relative position of the hole(s) or gunshot residue is recorded.

- 7.1.3. Perform macroscopic examinations using a stereo zoom binocular microscope with appropriate lighting. The examiner will be looking for various types of relevant physical effects and residues.
- 7.1.4. Indicative of/consistent with the discharge of a firearm:
 - 7.1.4.1. Vaporous lead (smoke).
 - 7.1.4.2. Particulate lead shavings or solidified droplets.
 - 7.1.4.3. Unburned gunpowder.
 - 7.1.4.4. Melted, adhering gunpowder.
- 7.1.5. Indicative of/consistent with the passage of a bullet:
 - 7.1.5.1. A hole(s) in an item.
 - 7.1.5.2. A visible ring around the perimeter of the hole(s) (bullet wipe).
- 7.1.6. Indicative of/consistent with a contact shot or close to contact shot:
 - 7.1.6.1. Ripping, tearing.
 - 7.1.6.2. Burning, singeing.
 - 7.1.6.3. Melted synthetic fibers.
 - 7.1.6.4. Heavy vaporous lead residues (smoke).
- 7.2. Chemical Tests
 - 7.2.1. After completion of the macroscopic examinations, certain chemically specific, chromophoric tests are conducted for the various types of gunshot residue. The initial test: the Modified Griess Test for Nitrite Residue, is directed toward the detection of deposits of nitrite compounds from burned or partially burned gunpowder around a suspected bullet hole(s) or patterns of suspected shot pellet holes.
 - 7.2.2. The Sodium Rhodizonate Test for Lead Residues procedure is directed towards the detection of any type of lead residue which might be present. This would include vaporous lead (smoke) usually associated with closer ranges, particulate lead and *bullet wipe*, a ring-shaped deposit often found around the perimeter of a bullet hole.
 - 7.2.3. The results of these tests will be reflected in the examination notes. The Chemical Control Data table in the Gun Shot Residue worksheet must be completed.
- 7.3. Modified Griess Test for Nitrite Residues
 - 7.3.1. Preparation of Nitrite Test Swabs
 - 7.3.1.1. Prepare a solution of 0.6 grams of sodium nitrite in 100 mL of distilled water in a beaker.

7.3.1.2. Divide the package of cotton swabs into equal amounts. Soak half of the cotton-tipped ends in the nitrite solution until saturated. Proper procedures for the disposal of the chemicals used are outlined in the Hazardous Waste Disposal section in the FEU Chemical Preparation Log Book.

7.3.1.3. Set the swabs aside to dry in the fume hood. Once dry, store in a labeled re-sealable bag. These will serve as the positive control. There is no known limit to the shelf life of these swabs.

7.3.1.4. Retain the non-treated swabs in a re-sealable bag. These swabs are used for the negative control.

7.3.2. Preparation of Reagents and Test Media

7.3.2.1. The following instructions apply to the preparation of the reagents and test media for use in the Modified Griess Test for nitrite residues. The desensitized photo paper mentioned below is simply photographic print paper which no longer bears light-sensitive silver salts in its surface emulsion. Validated inkjet photo paper (or Quantofix nitrite sheets) may be used in place of desensitized photo paper. Proper procedures for the disposal of the chemicals used are outlined in the Hazardous Waste Disposal section in the FEU Chemical Preparation Log Book.

7.3.3. Processing of Previously Desensitized Photographic Paper (Griess test paper)

7.3.3.1. Prepare a solution of 0.5 grams of sulphanilic acid in 100mL of distilled water in a beaker.

7.3.3.2. Prepare a solution of 0.28 grams of alpha-naphthol in 100mL of methanol in a beaker.

7.3.3.3. Combine the solution from step 2 into the solution from step 1 in a beaker.

7.3.3.4. Pour the combined solution into a non-reactive photo processing tray and briefly dip pre-cut sheets of the photographic paper into the tray. Submerge the sheets completely. Remove them and let air dry.

Note: As a substitute for photographic paper, ordinary laboratory filter paper may be processed in the same manner.

7.3.3.5. Set the sheets aside to dry. Dispose of any remaining solution as outlined in the Hazardous Waste Disposal section in the FEU Chemical Preparation Log Book.

7.3.4. Preparation of a 15% Acetic Acid Solution

7.3.4.1. Combine 150mL of glacial acetic acid with 850mL of distilled water. Gently pour the acid into the water to avoid the potential spattering of undiluted acid.

7.3.4.2. Store and label the solution in a properly sealed container with the identity of the reagent, date of preparation or lot number and the preparer's initials.

Note: All examiners will ensure that any original record (e.g. positive or negative) generated through GSR testing is complete and photographed prior to the destruction or disposition of the original record.

7.4. Procedure for the Modified Griess Test

7.4.1. To ensure desensitized and chemically treated photographic paper or filter paper is functioning properly; test the four corners of the emulsion-coated side with a positive control. This is accomplished by saturating a nitrite test swab (positive control) in a small amount of 15% acetic acid solution and dabbing the four corners of the paper. An orange color will appear at each corner, confirming sensitivity. The results will be recorded in the examination notes.

7.4.2. For a negative control, repeat the above step, but use clean, non-nitrite treated acetic acid-saturated test swabs. Ensure that this test follows the positive control test, and that there is sufficient physical separation between test marks to avoid bleeding from one mark to the other. The results will be recorded in the examination notes.

7.4.3. Place the evidence/questioned item or known-distance test questioned side down on the emulsion-coated side of the treated photographic paper. Index such things, i.e. seams, button holes, rips, pockets, suspected bullet holes, tears, cuts, etc., for possible future reference by marking with a pencil. DO NOT USE INK at this point because it may transfer back onto the tested item.

7.4.4. Soak a piece of cheesecloth in the 15% acetic acid solution (in a large beaker) and wring out to dispose of excess liquid. Place the cheesecloth on the evidence/questioned item or known-distance test as the third layer of the "sandwich". Press the "sandwich" with a hot iron. On many irons, the setting for "cotton" is appropriate.

7.4.5. Discard the cheesecloth into the biohazard container. Separate the evidence/questioned item or known-distance test-firing from the photographic paper.

7.4.6. When dry, the photographic paper will be marked appropriately. (Note: photographic paper is considered secondary evidence and will be marked with the lab #, Item #, defect # being processed, date and initial of the examiner. Retain any photographic paper showing positive results as part

of the raw data for inclusion in your notes. Test media relating to negative results will be photographed and need NOT be retained.

7.4.7. The Modified Griess Test Using Treated Filter Paper

- 7.4.7.1. Treat the filter paper in the same solutions used for treating the photographic paper. Allow it to dry. (See Processing of Previously Desensitized Paper).
- 7.4.7.2. Place the filter paper on the questioned surface. Test for nitrite sensitivity using positive and negative controls.
- 7.4.7.3. Process the filter paper using ONE of the following methods in the fume hood:
 - 7.4.7.4. Spray the filter paper with 15% acetic solution until very damp. Cover with two or three additional layers of the filter paper and iron until dry.
 - 7.4.7.5. Saturate a piece of cheesecloth in the 15% acetic acid solution and wring out. Place the cheesecloth over the filter paper and apply a hot iron.
- 7.4.7.6. Separate the test media and check results.
- 7.4.7.7. When dry, mark and preserve results for retention as in the above procedure.

7.5. Procedure for a Reverse Modified Griess Test

- 7.5.1. This test is used for thick or otherwise non-porous materials through which the acetic acid solution "Steam" will not penetrate.
 - 7.5.1.1. Tape a piece of filter paper or other appropriate nitrate-free substitute to the back of the piece of desensitized and treated photographic paper. Test as in the above procedures using nitrite test swabs.
 - 7.5.1.2. Place the photographic paper emulsion side down on the questioned surface and use a pencil to index seams, buttons, suspected bullet holes, pockets, rips, tears, cuts, etc., for possible future courtroom reference.
 - 7.5.1.3. Wipe the emulsion-coated side of the photographic paper with a piece of cheesecloth saturated with a 15 % acetic acid solution. Apply the solution to the entire surface, but lightly. Too much will cause indistinct or hazy results due to pigment migration.
 - 7.5.1.4. Immediately place the photographic paper emulsion side down on the questioned surface. Apply a hot iron to the back of the photographic paper until dry. Note that the back was previously covered by filter paper or an appropriate substitute; otherwise the paper may stick to the iron.

7.5.1.5. Separate the photographic paper and the questioned item. Any orange indications on the photographic paper are the result of the presence of nitrite residues.

7.5.1.6. When dry, mark and preserve any positive results for retention as in the previously described normal Modified Griess Test.

7.6. Sodium Rhodizonate Test for Lead Residues

7.6.1. Preparation of Reagents and Test Media

7.6.1.1. Storage of prepared chemicals and test media will be such that contamination is not possible. Storage containers will be kept sealed until the contents are needed. Fractions or multiples of the weights and volumes indicated may be used as appropriate to the amount of work to be done.

7.6.2. Preparation of the Sodium Rhodizonate Solution

7.6.2.1. Place a small amount of sodium rhodizonate in a beaker and add sufficient distilled water until the solution turns the color of dark tea. The solution is saturated if slight sediment is noted on the bottom of the beaker after stirring with a clean glass stirring rod. Make only enough solution for immediate use.

7.6.3. Preparation of 2.8 pH buffer Solution

7.6.3.1. Weigh 120 grains of sodium bitartrate in a weigh boat.

7.6.3.2. Weigh 90 grains of tartaric acid in another weigh boat.

7.6.3.3. Fill a beaker with 400 mL of distilled water.

7.6.3.4. Add sodium bitartrate to the distilled water and stir slowly with a glass stirring rod.

7.6.3.5. Add tartaric acid to bitartrate solution and slowly stir with a glass stirring rod.

7.6.3.6. Place a magnetic stir bar inside of the beaker and set the beaker on the magnetic stir plate for 10 minutes or until solute has dissolved.

7.6.3.7. Remove the stir bar and pour the contents of the beaker into a clean glass reagent bottle.

7.6.3.8. Label the bottle with the name of the reagent, batch number (date made and preparer's initials), and expiration date (one year from preparation date). Enter the appropriate information into the chemical preparation log.

7.6.4. Preparation of the Dilute (5%) Hydrochloric Acid Solution

7.6.4.1. Pour 50mL of Hydrochloric acid into a 100mL graduated cylinder.

7.6.4.2. Pour 950mL of distilled water into a 1000mL graduated cylinder.

- 7.6.4.3. Pour the distilled water into a 2000mL beaker.
- 7.6.4.4. Pour the 50mL of Hydrochloric acid into the beaker of water slowly by slightly tilting the beaker of water and letting the acid run down the interior of the beaker.
- 7.6.4.5. Stir slowly with a glass stirring rod.
- 7.6.4.6. Pour the solution into an appropriate storage container. Label the container with the reagent name, batch number (date made and preparer's initials), and expiration date (one year from preparation date). Enter the appropriate information into the FEU chemical preparation log.
- 7.6.5. Preparation of a 15% Acetic Acid Solution
 - 7.6.5.1. Combine 150mL of glacial acetic acid with 850mL of distilled water. Gently pour the acid into the water to avoid the potential spattering of undiluted acid.
 - 7.6.5.2. Store and label the solution in a properly sealed container with the name of the reagent, date and the preparer's initials.
 - 7.6.5.3. Enter information into the FEU chemical preparation log.
- 7.6.6. Preparation of Controls
 - 7.6.6.1. Positive Control: A lead bullet from the FEU ammunition room will be wiped across a piece of test material and the material then processed for the expected blue-violet reaction. The results will be recorded in the examination notes. Gloves must be changed after performing this positive control.
 - 7.6.6.2. Negative Control: Observing the absence of any blue-light color development on the non-wiped portions of the test material is sufficient for a negative control. The results will be recorded in the examination notes.
- 7.6.7. Direct Application to Items of Evidence
 - 7.6.7.1. Spray the appropriate area of the questioned item with the saturated solution of sodium rhodizonate.
 - 7.6.7.2. Spray the same area of the questioned item with the tartaric acid/sodium bitartrate buffer solution. This solution will eliminate the general yellow background color caused by the sodium rhodizonate and will establish a local pH of 2.8, turning any lead, as well as other metals that may be present, a pink color.
 - 7.6.7.3. Spray the same area with dilute hydrochloric acid solution. The presence of lead is specifically determined whenever the previous pink color fades out and leaves a blue-violet color in its place; this indicates lead and only lead. Be very aware that a positive (blue-

violet) result may abruptly fade. Note the results immediately after applying the dilute hydrochloric acid solution.

7.7. The Reverse Transfer Method for Dark-Colored Items (The Bashinski Transfer Method)

7.7.1. Place a piece of filter paper over the appropriate area of the questioned item in the fume hood.

7.7.2. Index the filter paper relative to the garment or other item to indicate the location of such things as suspected bullet holes, seams, buttons, button holes, pockets, rips, and tears. DO NOT USE INK at this point because it may transfer back onto the tested item.

7.7.3. Uniformly dampen the filter paper on the questioned item by spraying with 15% solution of glacial acetic acid.

7.7.4. Cover the dampened filter paper with several layers of dry filter paper. Apply a hot iron to the filter paper and iron until the paper is dry.

7.7.5. Remove the filter paper which was in direct contact with the evidence item, and process it using steps in (Direct Application to Items of Evidence) above. Note that any positive (blue-violet) indications are a mirror image of the deposition on the questioned item.

7.7.6. Prompt note-taking is essential in that sometimes the color can fade rapidly and unpredictably. Photograph any positive results. When dry, filter paper exhibiting positive results will be properly marked in ink for future identification, and retained. All results will be documented in the examination notes.

7.7.7. The Standard Transfer Method

7.7.7.1. Process the questioned item by following all steps in (Direct Application to Items of Evidence) above.

7.7.7.2. Blot the appropriate area of the questioned item using untreated filter paper.

7.7.7.3. Note any positive results. Such transfers usually reflect positive results which are very vague and indistinct in form.

7.7.7.4. Prompt note-taking is essential in that sometimes the color can fade rapidly and unpredictably. Photograph any positive results. When dry, filter paper exhibiting positive results will be properly marked in ink for future identification, and retained. All results will be documented in the examination notes.

7.8. Interpretation of Results

7.8.1. Gunshot residue distance determinations are a result of residues detected on an item(s) of evidence. The absence of residues is not a basis for expressing a distance determination. The results of the Sodium

Rhodizonate Test will be consistent with the results of the Modified Griess Test at a particular muzzle-to-target distance and with any physical effects present.

- 7.8.2. The Contact Shot or Close to Contact Shot: A contact shot or close to contact shot is based on the presence of very characteristic ripping and tearing of an item, the burning and singeing of cloth, the melting of synthetic fibers, and the heavy vaporous lead (smoke) deposits around the suspected bullet hole(s).
- 7.8.3. Nitrite Residues: With increases in muzzle-to-target distances, patterns of detectable nitrite residues around a suspect bullet hole(s) vary in size and density. When a pattern of nitrite deposits is found, it is possible to reproduce this pattern using the submitted firearm and like ammunition in combination. When only scattered nitrite residues are found, it is possible to find maximum distance to which such residues are deposited, using the submitted firearm and like ammunition in combination.
- 7.8.4. Vaporous Lead/Lead Residues: Vaporous lead deposits are characteristically deposited at close ranges and can be chemically detectable utilizing the Sodium Rhodizonate Test. Such residues are produced only if to a particular distance, which is determined utilizing the suspect firearm and like ammunition in known-distance tests. Lead *bullet wipe* is consistent with the passage of a bullet(s) and cannot determine distance.
- 7.8.5. Known Distance Test
 - 7.8.5.1. When reproducing residue patterns detected on evidentiary items, it is essential that the suspect firearm and like ammunition be used in the known-distance test. Patterns of residue will vary with changes in or to ammunition, barrel length, caliber, and powder charge.
 - 7.8.5.2. For most situations, white cotton twill cloth is suitable as a test target media. However, there may be instances where the characteristics of the evidence item are unusual enough to avoid meaningful test patterns with the cotton twill cloth. In such cases, it may be necessary to duplicate the evidence of material, or to utilize a portion of the evidence item for firing known-distance tests.
- 7.8.6. When certain types of residues are found, it is necessary to find maximum distance to which these residues are projected from a firearm. A procedure in these instances is to gather data that can be used to establish the distance at which the particular residue is always found, and the distance at which it is not found in known-distance tests. This forms a bracket for the maximum distance situation for a particular type of residue.

7.9. Range of Conclusions

7.9.1. Chemical Tests

7.9.1.1. The chemical tests typically yield three conclusions; Present, Not Present or undetermined.

7.9.2. Distance Determination

7.9.2.1. Based on the aforementioned chemical test conclusions a range may or may not be established for a muzzle to target distance.

7.9.2.2. The conclusions must be supported by the results and documented in an examiner's notes in the form of narratives, sketches and/or photographs.

7.10. Verification of Results

7.10.1. All detections of gunshot residues will be verified by a qualified examiner. The results will be documented in the LIMS verification worksheet.

7.10.2. There are two methods of verification:

7.10.2.1 The evidence is transferred in LIMS to the verifying examiner.

7.10.2.2 A transfer in LIMS is not required if the reporting examiner is present.

7.10.3. The reporting examiner will review the completed verification to ensure that there are no discrepancies in results and the results are documented correctly.

7.10.4. Any disagreements of results must be discussed between the reporting and verifying examiners. Both examiners must demonstrate the manner in which they reached their conclusions. The range of a target to distance can be increased or decreased if it is substantiated.

7.10.5. If an agreement of results is not reached, the FSL Quality Assurance specialist and FEU Management will be notified in writing, and review the results and objective evidence provided by examiners. Any evidence in question will be reviewed. A third analyst may be used to independently review the results, and the final decision will be made by the FEU Management or designee.

7.10.6. All verification results will be resolved before a report can be issued.

8. Sampling

8.1. Not applicable

9. Calculations

9.1. Not applicable

10. Uncertainty of Measurement

10.1. Not applicable

11. Limitations

- 11.1. The Modified Griess test is not appropriate for the detection of purely nitrate compounds, such as unburned smokeless powder. A practical reality, however, is that unburned powder particles (nitrates) are commonly coated with burned powder residues (nitrites) and positive reactions take place. The Modified Griess test yields results for nitrite residues regardless of whether these are in fact gunshot residues.
- 11.2. The Sodium Rhodizonate test yield results for the presence of lead regardless of whether these are related to the discharge of a firearm or the passage of a bullet.
- 11.3. Distance determinations reached as a result of gunshot residue examinations must be based on residues found to be present, not on the absence of residues.
- 11.4. Shooting events are dynamic and often complicated. Gunshot residue patterns together with chemical test results should be weighted accordingly to the scenario and the condition of the evidence. Without the firearm and like ammunition present the distance examination determination cannot be performed.

12. Documentation

- 12.1. FEU Chemical Preparation Log
- 12.2. Gunshot Residue worksheet

13. References

- 13.1. SWGGUN guidelines for Gunshot Residue Distance Determination (Revision 4/13/13)
- 13.2. Federal Bureau of Investigation Gunpowder and Gunshot Residue Manual (Current Version)
- 13.3. Beddow, et al. Quantofix Nitrite Sheet Validation for Use within the Modified Griess Test: A Technical Report. AFTE Journal 51 (1), 2019.
- 13.4. FSL Quality Assurance Manual (Current Version)
- 13.5. DFS Departmental Operations Manuals (Current Versions)
- 13.6. FSL Laboratory Operations Manuals (Current Versions)

13.7. FEU-LIMS-05 LIMS Guide to Gun Shot Residue (Current Version)

13.8. FEU Report Writing and Distribution (Current Version)

13.9. FEU Preparation and Storage of Reagents (Current Version)

Appendix A

FIREARM SAFETY

The following protocols must be adhered to when handling any firearms:

- 1.1 Wear additional PPE (eye & ear protection) when discharging a firearm.
- 1.2 Treat every firearm as though it is loaded until you verify that it is not.
- 1.3 Point the muzzle in a safe direction.
- 1.4 Keep finger off the trigger and outside the trigger guard until ready to fire.
- 1.5 Always ensure target and backstop.
- 1.6 When in doubt about a safety issue, consult with a senior examiner or FEU Management before starting a procedure.
- 1.7 When transporting firearms throughout the laboratory, employ a mechanism to visually ensure that the chamber is empty and/or the action is open. Acceptable methods are zip tie, empty chamber indicator, or locking the chamber in the open position if possible. Do not transport firearms with magazines inserted.
- 1.8 Ensure that the barrel and chamber are clear of obstructions before firing.
- 1.9 When preparing to fire, do not close the action of the firearm on a live cartridge unless the muzzle is pointed downrange or placed securely in the tube of the water recovery tank.
- 1.10 Follow all safety instructions for the use of test firing equipment, to include the water recovery tank, firing range, remote firing device, or any other bullet recovery method.
- 1.11 If a loaded or potentially loaded firearm is submitted, take it immediately to the firing range and render it safe.
- 1.12 Use extreme caution when inspecting/examining firearms that are damaged or in poor condition.
- 1.13 Utilize the remote firing device for any circumstances in which there may be a potential safety concern in discharging the firearm.
- 1.14 Utilize primed cases or downloaded cartridges for any circumstances in which it may be unsafe to discharge commercial cartridges (discretionary).

- 1.15 Do not use cell phones in the intake area, tank room, or firing range.
- 1.16 Do not allow live ammunition in the testing area for trigger pull weight determinations. Any examination that involves live ammunition must occur only in the designated test fire areas.
- 1.17 Do not allow individuals in the intake area work space, tank room, or firing range unless involved in the testing process.
- 1.18 Ensure that all individuals in the tank room or firing range are wearing appropriate PPE before the firearm is discharged.
- 1.19 Additionally, FEU staff are encouraged, though not required, to participate in the DFS Medical Surveillance Program, for regular testing of lead levels.
- 1.20 All FEU personnel are required to notify another analyst (or FEU Management) in FEU prior to and after test firing is performed.
- 1.21 In the absence of another analyst and FEU Management, appropriate personnel (i.e. DGS, MPD) should be notified prior to and after test firing is performed.
- 1.22 At any time, if the firearm that is required to be test fired is potentially malfunctioning, another analyst or appropriate personnel is required to be in the room with the analyst performing the test firing.